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Modeling Pilot Plant Performance of an Absorber with Aqueous Piperazine

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Abstract

Pilot plant data for CO_2 capture with 8 m piperazine (PZ) were reconciled with an absorber model in Aspen Plus® using quantified error in pilot plant input data and a global correction to absorber performance parameters. Four global corrections were applied independently to adjust: interfacial area, liquid side mass transfer coefficient, solvent CO_2 content, or solvent piperazine (PZ) content. Each of the four cases resulted in a reconciled model with pilot plant data and provides a potential route to quantifying and correcting measurement and experimental error as well as enhancing understanding of real absorber performance. The modified absorber model was then used to quantify the performance improvement due to implementation of a spray nozzle in the absorber intercooling loop. The spray nozzle added the equivalent of 7% to 20% more packing to the column as a function of the flow rate through the nozzle.

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*Keywords:*absorber; pilot plant; piperazine; data reconciliation; mass transfer; interfacial area; spray nozzle

1. Introduction

Pilot plant operations for CO_2 capture using amine solvents provide mass and energy balance data to validate thermodynamic, kinetic, and mass transfer models built using experimental data. In October 2011, the pilot plant at the University of Texas at Austin Separations Research Program (SRP) was operated with 8 molal (m) piperazine (PZ) in an intercooled absorber with solvent return via a spray nozzle. This paper will present validation results of an absorber model built using Aspen Plus®

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RateSep™, a thermodynamic and kinetic framework developed by Frailie [1], and mass transfer and area models developed by Wang [2]. The reconciled model was used to quantify the performance benefit of spray nozzle implementation in the intercooling loop.

2. Pilot Plant Overview: October 2011 Campaign

The pilot plant at SRP includes an absorber, stripper, and 2-stage flash skid used in CO_2 capture trials. The October 2011 campaign used the 2-stage flash skid for stripping; see Madan et al. [3] and Walters et al. [9] for details regarding the stripping section and Chen et al. [4] for an overview of the SRP pilot plant equipment, operations, and overall performance results. The absorber configuration for the October 2011 campaign is depicted in Figure 1.

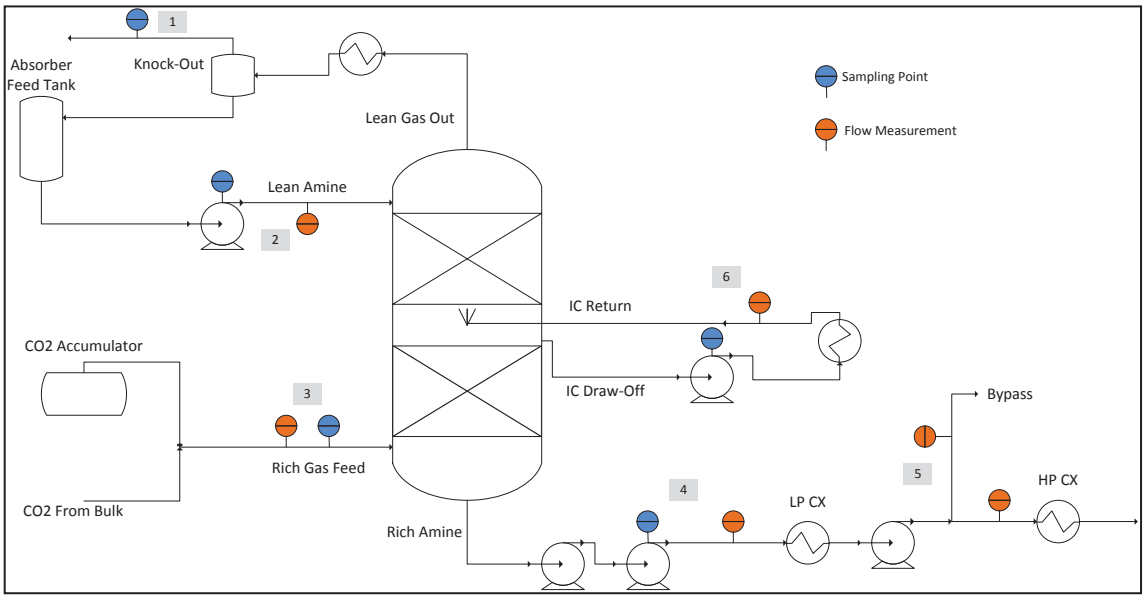

Figure 1 Absorber PFD with flow and sampling points, SRP October 2011 Campaign

The campaign included two absorber-specific equipment modifications from previous campaigns. First, the campaign was the first trial of a 350 series packing (specific area of 350 m^2/m^3) with a 70° corrugation angle used in CO_2 capture applications with 8 m PZ. Second, the intercooling loop included a spray nozzle configured to feed the intercooled solvent into the bottom of the upper section of packing (see Figure 1).

Table 1 provides an overview of equipment and operating specifications relevant to absorber performance and modeling for the 11 runs of the October 2011 campaign.

Table 1. Overview of Equipment and Operating Specifications, October 2011 Pilot Plant Campaign

Equipment Specifications		Operating Specifications	
Column		**Solvent (PZ)**	
Inner Diameter (m)	0.43	*Concentration(m)*	8
Packing (Structured)		*Liquid Rate (GPM)*	11 - 22
Height (m)	6.1 (2 beds x 3.05)	*Lean Loading (mols CO_2/mols alkalinity)*	0.24-0.26
Specific Area (m^2/m^3)	350	**Feed Gas**	
Corrugation Angle (°)	70	*CO_2 (mol %)*	12%
Material	Stainless	*Gas Rate (ACFM)*	350 - 675

3. Modeling Framework

The model for the PZ solvent and absorber equipment configurations was developed in Aspen Plus® RateSep™. The thermodynamic model for the PZ-H_2O-CO_2 system was developed from experimental CO_2 solubility, heat capacity, speciation, and amine volatility data by regression of Gibbs free energy, enthalpy, heat capacity and activity coefficient parameters within the electrolyte non-random two liquid (e-NRTL) framework in Aspen Plus®. For a detailed description of the sequential regression methodology used in the development of the PZ thermodynamic model, see Frailie et al. [5]; for details regarding the most recent version of the PZ model, as implemented in the current work, see Frailie [1].

Mass transfer and area models were developed by Wang [2] via regression of experimental data from a pilot scale column with a variety of random and structured packings. The area model developed by Wang is a modification of a model developed by Tsai [6] (see Tsai for full theoretical and experimental details of the area model). The mass transfer model is discussed in additional detail in subsequent sections.

4. Data Reconciliation Methodology

The data reconciliation process consists of four broad steps:

- Quantification of error in pilot plant measurements;
- Identification of global adjustable parameters for data reconciliation;
- Reconciliation of pilot plant mass and energy balances for runs *without spray nozzle* implementation;
- Isolation of spray nozzle performance by application of global adjustments from step 3 with independent variation of spray nozzle mass transfer area for each run *with spray nozzle implementation*.

Each of the steps is discussed in further detail in the subsequent sections. The data reconciliation tool in Aspen Plus® used to implement the process described in the steps minimizes the objective function described by Equation 1 to perform a maximum likelihood (errors in variables) regression on the pilot plant data.

$$\underset{v}{Min} \sum_{i=1}^{N} \left(\frac{(Measured_i - Model_i)}{\sigma_{measured,i}} \right)^2 \quad (1)$$

Where:

$\boldsymbol{Measured_i}$ are the pilot plant measured input or result parameters,
$\boldsymbol{Model_i}$ are the process model predicted input or result parameters,
$\boldsymbol{\sigma_{measured,i}}$ are the pilot plant calculated standard deviations for input and result parameters, and
$\boldsymbol{v}$ are the globally varied parameters.

4.1. Error Quantification

Measureable error in the pilot plant data was limited to random error quantified via repeated trials of analytical measurements and continuous data collection of inline measurements at steady state (flows, temperatures, etc.). Table 2 summarizes measurement techniques used for the sampling points around the absorber identified in Figure 1.

Table 2. Measurement Techniques used in October 2011 campaign

Measurement Location and ID	**Inline**	**Analytical**	
		CO_2 concentration	**PZ concentration**
Lean Amine/Mid-Column Amine (Point 2 and 6)	Micro Motion® Coriolis Flow Meters	Auto Titration Manual Titration Total Inorganic Carbon	Auto Titration Manual Titration
Rich Amine (Points 4 and 5)	Micro Motion® Coriolis Flow Meter	Auto Titration Manual Titration Total Inorganic Carbon	Auto Titration
Lean Gas (Point 1)	Rosemount® Annubar (Differential Pressure) Meter	Vaisala® GMT220 CO_2 Sensor (NDIR Sensor)	N/A
Rich Gas (Point 3)	Inferred from Inlet	Vaisala® GMT220 CO_2 Sensor (NDIR Sensor)	N/A
Column Temperatures (Throughout Column)	Rosemount® 68-Series RTD	N/A	N/A

Standard deviations were calculated for each measurement from the pilot plant and were propagated through calculations of derived values (e.g., component flow rates) with assumption of uncorrelated

random error (covariance terms are omitted). The results of the error analysis allowed verification of mass balance closure within random error in the process (discussed in results section) and provided a range for input variables used in data reconciliation.

4.2. Global Adjustment Parameters

Global adjustment parameters are user-defined model parameters which are adjusted uniformly for all of the experiments in a dataset (i.e., all of the runs in a pilot plant campaign) as part of the error minimization process described by Equation 1. For pilot plant campaigns, parameters were selected to reflect sources of potential systematic bias in data measurements and/or physical parameters linked to column performance. The choice of parameters will guide future pilot plant operations and experimental research. The four parameters selected for evaluation in the October 2011 campaign were mass transfer area, liquid-side mass transfer coefficient, lean solvent CO_2 content, and lean solvent PZ content.

4.2.1. Correction for Mass Transfer Area

The mass transfer area model originally developed by Tsai [6] from data for 9 different types of structured packing matched experimental data within 13%. The correction to the model-predicted area in the data reconciliation process indirectly accounts for, in part, the model error. In addition, liquid distribution issues (specifically, liquid flows along walls) can result in under-utilization of packing; Yin et al. demonstrated significant wall flow in a column of similar diameter to the SRP pilot plant column (0.5 m vs. 0.43 m) when utilizing random packing [7]. Thus the area factor can be used to represent model error and physical performance issues.

4.2.2. Correction for Liquid Side Mass Transfer Coefficient

The liquid side mass transfer model implemented in the reconciliation process is a simple empirical equation (Equation 2) designed to fit data collected by Wang on the 350 series packing with a viscosity correction to account for the use of a viscous solvent (PZ) instead of the water used in experimental mass transfer studies [2].

$$\frac{k_L}{D^{0.5}} = A * \left(\frac{U_{SL}}{a_p} \right)^B * \mu_L^{-0.5} \tag{2}$$

Where:

$\boldsymbol{k_L}$ is the liquid side mass transfer coefficient (m/s),
$\boldsymbol{D}$ is the binary diffusivity coefficient (m^2/s),
A and B are the regression parameters (A = 281 $kg/s^{0.637}/m^{0.726}$, B = 0.863 for the 350 series packing in this work),
$\boldsymbol{U_{SL}}$ is the superficial liquid velocity (m/s),
$\boldsymbol{a_p}$ is the specific area of the packing (m^2/m^3), and
$\boldsymbol{\mu_L}$ is the liquid viscosity (Pa-s).

The model is presented as a diffusion independent mass transfer coefficient (consistent with implementation in Aspen Plus®) with the assumption that the mass transfer coefficient has a square root dependence on diffusivity as predicted by penetration and surface renewal theories. The prediction of

diffusion coefficient is not considered in this work, but is developed as part of the thermodynamic framework of Frailie [1]. Equation 2 is not a generally applicable mass transfer model, but rather an empirical expression to represent the experimental data collected for the packing of interest (350 series packing in this work). The exponent on the viscosity term was derived from literature review since viscosity was not varied in the experimental data.

Physical mass transfer resistance in reactive absorption systems with fast (not instantaneous) reaction is not significant under certain conditions (pseudo-first order approximation with high Hatta number [8]). For the liquid side mass transfer model to be a meaningful parameter in the data reconciliation process, the predicted mass transfer coefficient must be low enough to limit diffusion of reactant and products to and from the interface (violating the pseudo-first order approximation), reducing the rate of CO_2 absorption. A sensitivity analysis of the mass transfer coefficient for representative pilot plant conditions (lean loading = 0.28, L/G = 4.3 mol/mol, spray intercooling) is summarized in Figure 2. The results depict two distinct regions of CO_2 mass transfer sensitivity to k_L; the prediction of Equation 2 (red, dashed line) falls in the region of high sensitivity (represented by the slope of the line or exponent on the trend line equation in Figure 2). Thus, current experimental data for the 350 series, 70° packing indicate that the physical mass transfer coefficient is important to overall mass transfer performance in the pilot plant system with 8 m PZ.

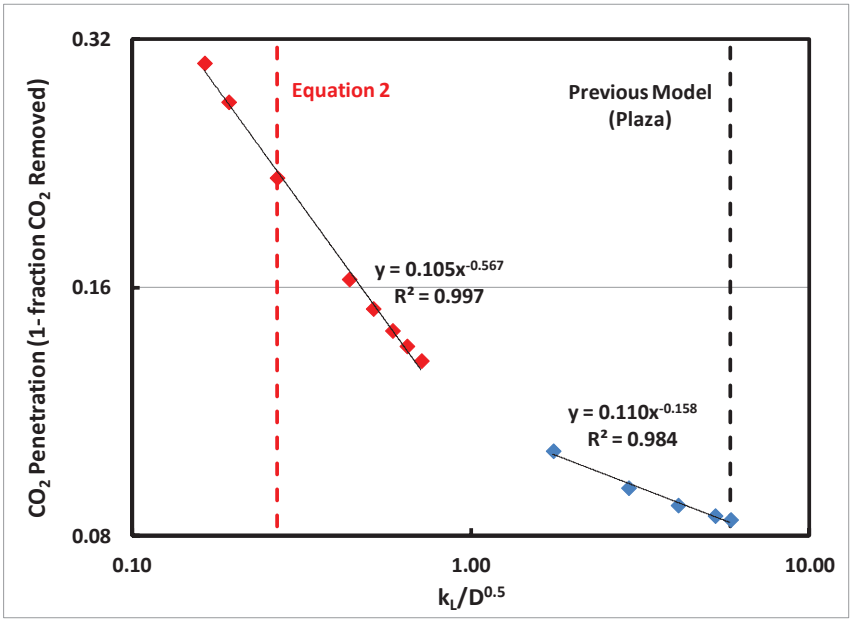

Figure 2 Sensitivity of CO_2 Absorption (Penetration) to changes in Diffusion-Independent Liquid Mass Transfer Coefficient. Dashed lines represent predictions by the mass transfer model presented in this work **(- -red)** in Equation 2 and the model used by Plaza [9] (**- - black)** in previous pilot plant analysis. All data in the plot reflect the following modeled case: lean loading = 0.27 mol CO_2/mol alkalinity, L/G = 4.3 mol/mol, and spray intercooling. Mass transfer coefficients are average values over the column.

Since the model parameters were regressed to fit the data for the packing used in this pilot plant campaign, the average deviation of the model from the experimental data was small (< 1%), but individual experimental points could vary from model predictions by as much as 20%. Therefore, this

model error may be represented in the reconciliation process by allowing a correction to the model predicted mass transfer coefficient. In addition, as with the interfacial area, local mass transfer coefficients can be adversely affected by liquid distribution in the column. If large portions of the packing do not receive sufficient liquid flow, the apparent mass transfer coefficient will drop correspondingly; local mass transfer behavior is not easily modeled in packed columns, and thus the correction to the model-predicted mass transfer is needed.

4.2.3. Corrections for CO_2 and PZ

Corrections for CO_2 and PZ component mass flows in the feed stream to the absorber were also implemented (each an independent parameter). The corrections were primarily introduced to represent potential measurement bias in titrations or flow measurements around the absorber. Mass balance reconciliation after random error quantification (section 4.1) showed that the CO_2 and PZ mass balances could not be simultaneously closed within the random error using the same titration and flow meter data sources. This inconsistency pointed to potential bias in either flow or titration data. In addition, work by Walters showed that CO_2 concentration predictions from inline density measurements were inconsistent with CO_2 titration data [9]. This evidence supports the need for a bias correction in the reconciliation process. Finally, work by Nielsen indicated evidence of degradation products in the pilot plant solvent [10]; if the PZ degraded over time, and this corresponds to a loss of alkalinity of the solvent, the modeled PZ concentration should be reduced to reflect reduced alkalinity.

4.3. Data Reconciliation and Spray Nozzle Performance

The pilot plant data and global adjustment parameters were used to reconcile model predicted performance with pilot plant measurements (Equation 1) for the three runs in the October 2011 campaign (runs 1, 8, and 10) that did not implement spray nozzle intercooling; this process identified values for the global adjustment parameters to be applied uniformly to the full campaign. With global parameters fixed, the spray nozzle was modeled as a packed section between the two primary beds of the column. The mass transfer area of the spray nozzle section was treated as an independent variable for each run with the spray nozzle activated and provided a measure of interfacial area generated by the spray without rigorous modeling for the spray. This two-step process identified global adjustments for the entire campaign and isolated the impact for the spray nozzle by run. Table 3 summarizes input, adjustment, and result parameters used in the data reconciliation.

Table 3. Data Reconciliation Parameter Summary

Input Parameters	Global Adjustment Parameters	Results
Lean Solvent - CO_2	Liquid Side Mass Transfer Coefficient (kL)	Rich Solvent Flow
Lean Solvent - PZ	Interfacial Area	Mid Column Loading[1]
Lean Solvent – H_2O	CO_2 Correction	Rich Loading[1]
Lean Solvent –Temperature/Pressure	PZ Correction	Rich Solvent Temperature
Rich Gas Composition[2]		Lean Gas Composition[2]
Rich Gas Temperature/Pressure		Lean Gas Temperature
Rich Gas Flow		Lean Gas Flow
Intercooling Temperature		
Column Pressure Drop		
Column Heat Loss[3]		

1. Mid-Column and Rich Loadings were represented by changes from lean loading
2. Rich and Lean gas compositions were used to calculate CO_2 Removal
3. Steady state heat loss calculated from column RTD measurements.

5. Previous PZ Campaigns and Data Reconciliation

Three previous campaigns were implemented using 8 m PZ at SRP. Plaza performed data reconciliation work on the 3 previous campaigns, using both an interfacial area modification and CO_2 modification to reconcile the pilot plant data with a previous version of the 8 m PZ model [11]. Table 4 provides a summary of absorber conditions and the results of Plaza. The significance of the CO_2 corrections cannot be isolated from the mass transfer area corrections since they were varied concurrently and are likely not independent; this is also reflected in the standard deviations around the corrections which indicate the corrections are not statistically different from 1(no correction) at a 95% confidence level (only the December 2010 area correction is significant). Nonetheless, the corrections provide a reference for comparison and range of expected corrections for the October 2011 campaign.

Table 4. Previous PZ Pilot Plant Campaign Operating Conditions and Reconciliation Results

Pilot Plant Campaigns		November 2009	September 2010	December 2010	October 2011*
Operating Conditions	Solvent	5 – 9 m PZ	8 m PZ	8 m PZ	8 m PZ
	Packing Type/Sp. Area(m^2/m^3)/Angle	Structured 205 X	Hybrid 250	Hybrid 250	Structured 350 Z
	Gas Rate (ACFM)	350	250-750	350-650	350-675
	Liquid Rate (GPM)	12-18	8-26	8-26	11-22
	Intercooling	No	Yes/No	Yes	Yes (with Spray)
Modified Model Parameters	Interfacial Area Factor	1.17 ± 0.15	1.02 ± 0.16	0.72 ± 0.13	See Results
	CO_2 Multiplier	1.05 ± 0.03	1.05 ± 0.03	1.06 ± 0.04	See Results
	# of Runs Evaluated	14	12	9	11

6. Results

Table 5 summarizes the global parameter sensitivity results of the first step of the reconciliation process (runs without the spray nozzle).

Table 5. Results of Independent Global Parameter Sensitivity Analysis with 95% confidence intervals

Parameter	Correction Factor	Upper 95%	Lower 95%
Interfacial Area	0.74	0.79	0.68
Liquid Side Mass Transfer Coefficient (k_L)	0.65	0.71	0.59
CO_2 Correction	**1.075**	**1.1**	**1.05**
PZ Correction	0.93	0.95	0.91

Each of the individual parameter corrections in Table 5 is statistically significant and reconciled the overall mass and energy balance for the 3 runs without the spray nozzle. The area and CO_2 corrections are similar to previous corrections by Plaza [11], though a combination of area and loading corrections was not needed to reconcile the data in this work (less severe correction than previous work). The CO_2 correction has corroborating data from the work of Madan in stripper reconciliation and Walters in dynamic pilot plant performance analysis [3,9]. Madan was able to reconcile the stripper section (two-stage flash) of the October 2011 campaign with a correction of 4.6% reported in this work [3]. Walters work showed that an average increase of 4.5% was required in CO_2 mole fractions to reconcile dynamic simulation around the stripping section [9]. The correction in this work is higher than those on the stripper

side, and may in reality be coupled with one of the other effects in represented in Table 5. Nonetheless, the CO_2 correction was selected as the global parameter to be applied to all spray nozzle runs.

Figure 3 through 6 show several important result parameters from the data reconciliation process using the CO_2 correction and spray nozzle mass transfer area variation. The reconciliation process matched all input and result variables within 95% confidence intervals (error bars in figures) with the exception of mid column loading (not shown). The single parameter approach used in this analysis is likely insufficient (underspecified) to fit the mid loading measurement, and a secondary parameter would be needed to account for relative mass transfer rates through the column.

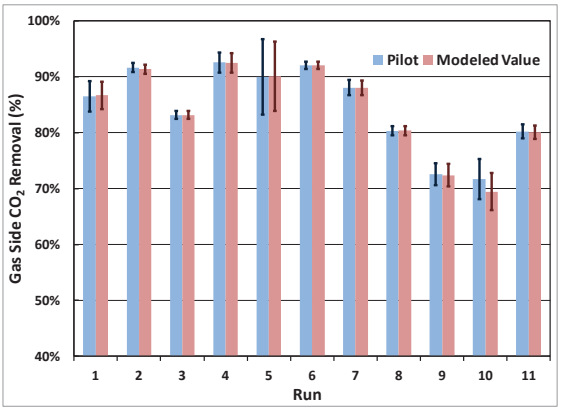

Figure 3 Gas Side CO_2 Removal by Pilot Plant Run, Measured **(blue)** vs. Model Predicted **(red)** (Error bars = 95% Confidence Intervals)

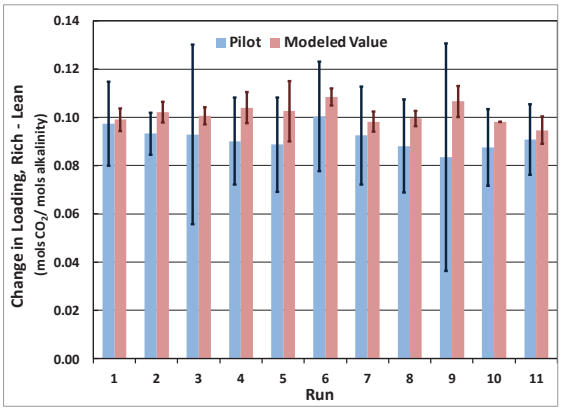

Figure 4 Loading Difference (Rich – Lean) by Pilot Plant Run, Measured **(blue)** vs. Model Predicted **(red)** (Error bars = 95% Confidence Intervals)

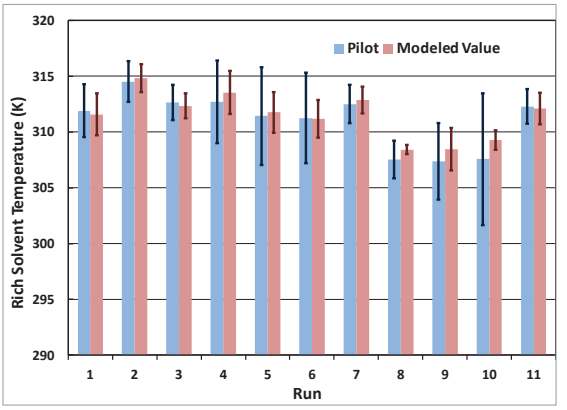

Figure 5 Rich Solvent Temperature by Pilot Plant Run, Measured **(blue)** vs. Model Predicted **(red)** (Error bars = 95% Confidence Intervals)

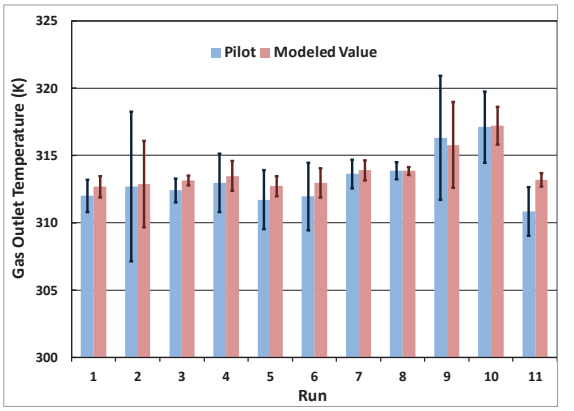

Figure 6 Gas Outlet Temperature by Pilot Plant Run, Measured **(blue)** vs. Model Predicted **(red)** (Error bars = 95% Confidence Intervals)

In addition to verification of the reconciliation process, the result parameters provide insight into the pilot plant operations. The rich to lean loading difference (Figure 4) shows a trend of over-prediction by the model (all 11 runs). Random process variations and measurement errors should yield pilot plant results on both sides of the model prediction; it is highly unlikely to find trends such as that in Figure 4 strictly via random variation. Reconciliation results for the rich solvent flow rate revealed an analogous trend; the rich solvent flow rate was shifted towards its lower bound in all but one run. The drop in solvent flow for a given CO_2 transfer leads to higher rich loading as observed in Figure 4. During the error quantification process of the pilot plant data (section 4.1), the choice of rich flow meter (point 4 vs. point 5 in Figure 1) altered the mass balance closure. An average of flow meter values was used in the reconciliation since this minimized the error in the mass balance; however, as the reconciliation results indicate, the rich solvent flow still shows an apparent bias that should be addressed in future runs.

Figure 7 and 8 provide representative temperature profiles for the column for runs with comparable operating conditions with and without the spray nozzle.

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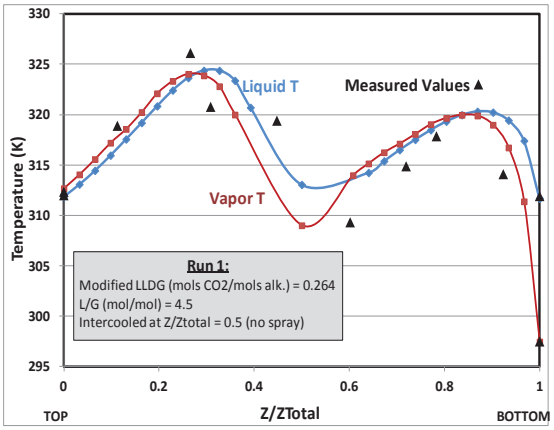

Figure 7 Absorber Column Temperature Profile, Run 1 (Intercooling with No Spray Return). Profile moves down the column from left to right.

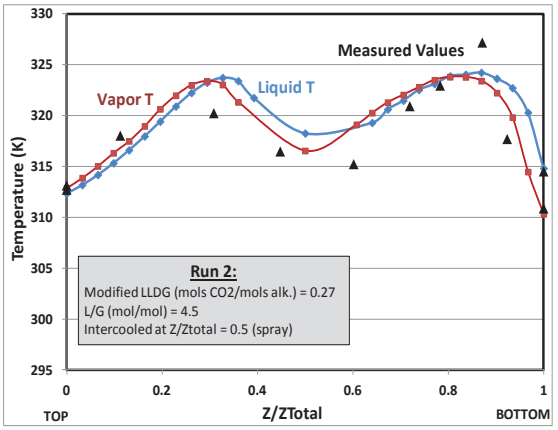

Figure 8 Absorber Column Temperature Profile, Run 2 (Spray Return for Intercooling). Profile moves down the column from left to right.

Despite the fact that the reconciliation process only matched inlet and outlet gas and solvent temperatures, the temperature profile behavior is largely captured by the model. For example, in run 2, the spray nozzle leads to recycle of solvent in the middle of the column (approaches a well-mixed section) and the temperature leaving the middle section of the column is higher than in run 1 where the intercooled solvent goes directly to the lower section of the column; this behavior is replicated well by the model. The model struggled to match the peak temperatures observed at the pilot plant; this may be a function of poor estimates of heat loss at these points or may provide insight into real temperature behavior in operation. These points may be candidates for multiple radial temperature measurements in future campaigns.

Finally, the predicted mass transfer area generated by the spray nozzle was quantified as an equivalent height of packing (Table 6). The predicted height was then correlated to the mass flow rate through the spray nozzle with the purpose of relating the kinetic energy in the flow stream to the mass transfer area generated from the nozzle (Figure 9).

Table 6. Predicted Spray Nozzle Mass Transfer Area (Equivalent Height of Packing) by Pilot Plant Run

Run	Spray Equivalent Height of Packing (m)	% Addition to Total Packing
2	**0.44**	**7%**
3	**0.58**	**10%**
4	**0.96**	**16%**
5	**0.57**	**9%**
6	**1.03**	**17%**
7	**1.09**	**18%**
9	**1.26**	**21%**
11	**0.88**	**14%**

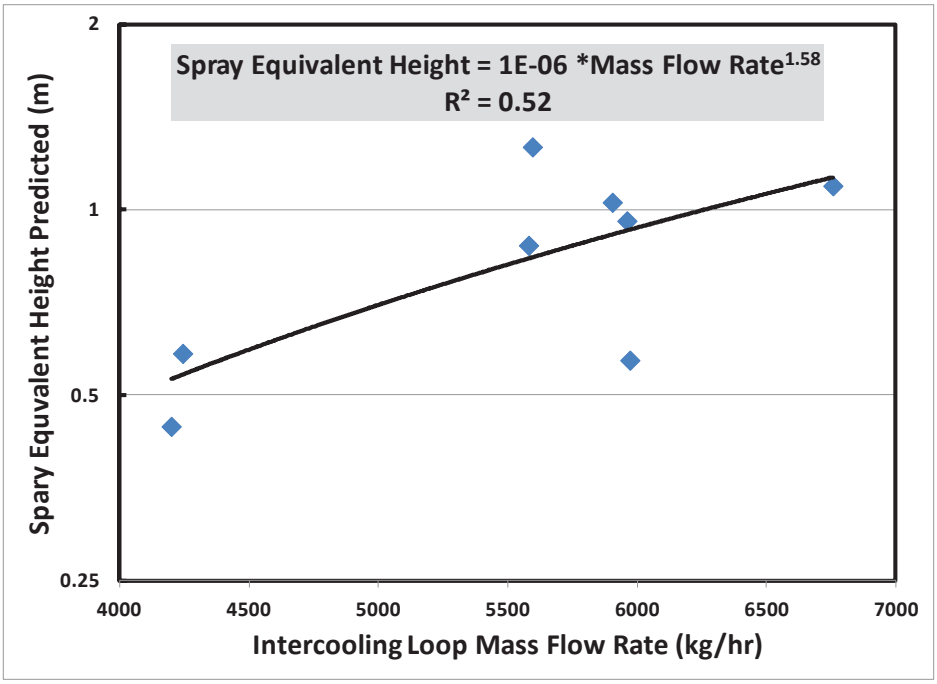

Figure 9 Prediction of mass transfer area produced by the spray nozzle on intercooled solvent return as a function of the solvent mass flow rate through the nozzle. Mass transfer area is reported as an equivalent height of packing (of 350 series packing used in the October 2011 campaign).

The data regarding spray nozzle performance can be used to perform an economic analysis of spray nozzle implementation (trade-off vs. packing) or as a measure of performance improvement in a retrofit application in an absorber column. In the case of the pilot plant, additional mass transfer area was generated where packing could not be added at the cost of a spray nozzle and pressure drop associated with the flow rates enumerated in Figure 9.

7. Conclusions

Pilot plant data reconciliation of mass and energy balances for an absorber column utilizing 8 m PZ were successfully completed with the independent implementation of 4 global adjustment parameters representing potential deviations in pilot absorber performance from model predictions.

- Interfacial Area Correction: A 26% decrease of the model predicted mass transfer area was required to reconcile the October 2011 campaign data with model predictions. The reduced performance compared to the model (and experimentally collected data) in the pilot column may indicate error in

the model at pilot plant conditions or issues with column operation such as liquid distribution. The result suggests the need for proper incorporation of experimental error into modeling efforts as well as reduction of model error by packing specific area measurements at pilot plant conditions to reduce error in the model. This objective should be balanced with the need for generalized area models that are applicable to a variety of packing and will inherently have more error than a packing-specific model. In addition, experiments quantifying the performance impact of varying liquid distribution would provide a valuable upgrade to the area models in use currently.

- Liquid Side Mass Transfer Coefficient: Sensitivity analysis of the mass transfer coefficient revealed that column performance is sensitive to the physical mass transfer coefficient. A 35% reduction of the nominal or model predicted mass transfer coefficient was required to reconcile pilot plant data. As with the mass transfer area, repeated experiments at pilot plant conditions could isolate the relevant experimental error in the mass transfer model and liquid distribution experiments could provide an additional important correction parameter. Finally, the viscosity dependence assumed in this work (-0.5 power) should be experimentally verified or updated for systems analogous to the 8 m PZ solvent as this is another source of potential uncertainty currently unaccounted for in the model.

- CO_2 Correction: An increase of 7.5% to CO_2 content (mole fraction) in the lean amine stream was required to achieve reconciliation between model and plant data. As noted, the correction to CO_2 is in part validated by stripper reconciliation work by Madan [3] and Walters [9] who found increases in CO_2 concentration of 4.6 and 4.7%, respectively. The correction in CO_2 concentration implies a bias in pilot plant measurement data (titrations and/or flow measurements); this potential for bias was supported by component mass balance closure problems during error quantification of pilot plant data. However, future campaigns will require re-calibration of flow meters and analysis of standards for analytical methods to quantify or eliminate the bias.

- PZ Correction: A 7% reduction to PZ content (mole fraction) in the lean amine stream was required to achieve reconciliation between model and plant data. As with CO_2, the correction may be explained by un-quantified measurement bias. However, work by Nielsen [10] points to the possibility of degradation of the PZ solvent over several campaigns which may ultimately lead to loss of alkalinity in the solvent. However, this effect has not been quantified in a way that can be modeled. The establishment of a baseline for the solvent condition based on Nielsen or the use of fresh solvent in future campaigns will allow the evaluation of modeled compared to actual solvent performance over time. Alongside analytical data collected with each campaign, changes in expected performance can be correlated to changes in the solvent.

Using the CO_2 model correction, the effect of spray nozzles was quantified in terms of the equivalent height of packing required to replicate spray nozzle performance improvement. The spray nozzle impact was equivalent to the addition of 7 to 20% additional packing to the column as configured in the October 2011 campaign. Further, the spray nozzle mass transfer area generated was correlated to the 1.5 power of the mass flow rate through the nozzle (and intercooling loop). This correlation provides the basis for economic evaluation of the spray nozzle configuration and identification of feasible conditions for operation.

Acknowledgements

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